SIMULTANEOUS DETERMINATION OF Rb AND Cs
BY RNAA METHOD

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A RNAA method has been developed for the simultaneous determination of Rb and Cs in geological samples. The method is based on precipitation with sodium tetraphenylborate followed by NaI(Tl) gamma spectrometry. Three mica dust particulate samples and two USGS standard rocks, BCR-1 and W-1 were analyzed. Dipercyamine (DPA) and 2-thienyl-trifluoroacetone (TTA) in nitrobenzene were also used for solvent extraction. The precipitation method is better than solvent extraction.

Introduction

The alkali metal concentrations in igneous and ultramafic rocks are of importance because of their role in the formation history. While Na and K are major and/or minor constituents, Rb and Cs are found at trace level, < 100 ppm, only. These act as indicator elements to elucidate the degree of geochemical fractionation. Neutron activation analysis (NAA) has been widely used for the determination of Rb and Cs in geological samples after ion exchange, chromatographic separations, co-precipitation, or chemical group separation methods.

Determination of rare alkali metals in samples of geochemical interest has been previously reported by SMALES and his coworkers using NAA with ion exchange separations. Later a procedure was described for the simultaneous determination of Rb and Cs in rock samples using a Ge(Li) detector. An ion exchange separation method using ammonium molybdophosphate (AMP) followed by precipitation has been developed. Several other methods have also been suggested for their determination. The INAA method for Cs in rocks even at low concentration quite often gives satisfactory results. However, the method is not suitable for Rb mainly because of low cross section (\( \sigma = 0.91 \text{ b} \)) and because the photopeak at 1077 keV due to the \(^{87}\text{Rb} \) interferes with the 1099 and 1120 keV \( \gamma \)-rays due to \(^{59}\text{Fe} \) and \(^{46}\text{Sc} \), respectively, unless a high resolution Ge(Li) detector is available. Existing
methods involve time-consuming ion exchange and chemical group separation schemes or use a Ge(Li) detector.

We have developed a procedure based on NAA followed by precipitation as tetraphenylborate and counting with a scintillation detector. This is advantageous because Rb and Cs can be determined simultaneously using a NaI(Tl) detector coupled to a single channel analyzer. We have also used dipicrylamine (DPA) and 2-thenoyltrifluoroacetone (TTA) as extraction reagents for the determination. Three mica dust particulates and two USGS standards were analyzed. A comparative performance of the reagents is described.

Experimental

Sample preparation, irradiation and counting

Three mica dust particulate samples were collected from the mica mines and factory area from the eastern part of India (Bihar). About 100 mg each of USGS rocks BCR-1 and W-1, standards and samples were sealed in high purity quartz ampoules and irradiated for 4 weeks at a thermal neutron flux of $2 \times 10^{12}$ n cm$^{-2}$ s$^{-1}$ in the APSARA reactor of the Bhabha Atomic Research Centre Trombay, Bombay. Chemically processed samples were counted for gamma activities of $^{86}$Rb and $^{134}$Cs on a well – type $2'' \times 1.75''$ NaI(Tl) detector coupled with a single channel analyzer. The nuclear data for the two elements studied here are given in Table 1.

<table>
<thead>
<tr>
<th>Element</th>
<th>Nuclear Reaction</th>
<th>Isotopic abundance, %</th>
<th>Half-life</th>
<th>Cross section, barn</th>
<th>$E_\gamma$, keV</th>
</tr>
</thead>
<tbody>
<tr>
<td>Rb</td>
<td>$^{85}$Rb(n, $\gamma$)$^{86}$Rb</td>
<td>72.15</td>
<td>18.7 d</td>
<td>0.91</td>
<td>1077</td>
</tr>
<tr>
<td>Cs</td>
<td>$^{133}$Cs(n, $\gamma$)$^{134}$Cs</td>
<td>100</td>
<td>2.05 y</td>
<td>30.0</td>
<td>605</td>
</tr>
</tbody>
</table>

Reagents and solutions

All the chemicals were of AR, guaranteed or of high purity grade and used as such. Sodium tetraphenylborate (0.2M), carrier solutions for Rb and Cs as RbCl or CsCl (5 mg Rb or Cs/ml), sodium salt of EDTA (4%), acetic acid (0.1M) and